



Docket No. 71624 CCD

IN THE UNITED STATES
PATENT AND TRADEMARK OFFICE

APPLICATION NO : 10/723,966
APPLICANT : DIONNE, Martin; et al
FILED : November 26, 2003
TITLE : STABILIZERS FOR TITANIUM DIBORIDE-
CONTAINING CATHODE STRUCTURES
ART UNIT : 1742
EXAMINER : LEADER, William T.

DECLARATION UNDER 37 C.F.R. § 1.132

I, Dr. Pierre-Yves BRISSON, declare that:

1. I have been a full-time employee of Alcan International Limited, the assignee of the present application, since January 1, 2006, and I was employed under contract to the same company from June 2005 until completion of my Ph.D. My current position with the company is Research Scientist in the Strategic Research Group working on materials for wettable cathodes.
2. I have a Ph.D. in Chemical Engineering from the Université de Sherbrooke, Québec, Canada. The subject of my thesis was the study of sodium and bath penetration mechanisms within the carbon cathode of the aluminum electrolysis cell. Consequently, the subject of my Ph.D. is relevant to studies on deterioration of carbon materials (including those containing titanium diboride) in electrolysis cells. I also hold the degree of M.Sc.A., Metallurgical Engineering, from École Polytechnique de Montréal, 2002. The subject of my master's thesis was the understanding of the relation between the microstructure of refractory materials and their mechanical behaviour at high temperatures (1200 to 1400°C). I also hold the degree of B.Eng. Materials Sciences and Engineering, from École Polytechnique de Montréal, 2002.
3. I performed the following experiment during June and July 2006 in Alcan's research and development centre in Jonquière, Québec in order to assess the performance differences between materials with TiB_2 only versus materials containing partial substitution of TiB_2 by a mixture of the precursor oxides. The experiment involved a laboratory scale electrolysis cell in which the cathode material was formed into a cylindrically shaped cathode and was rotated in the vertical plane between two anodes set about 2.5 cm from the cathode surface. The test equipment is illustrated in Figs. 1 and 2 below.



Figure 1 shows the apparatus. In this drawing, the anode and the cathode are withdrawn from the bath which lies underneath. The current is transmitted to the rotating shaft through a special connector ("slip ring"). Figure 2 shows schematics (a vertical cross-section and a top plan view) of the cathode/anode arrangement during electrolysis in the bath.

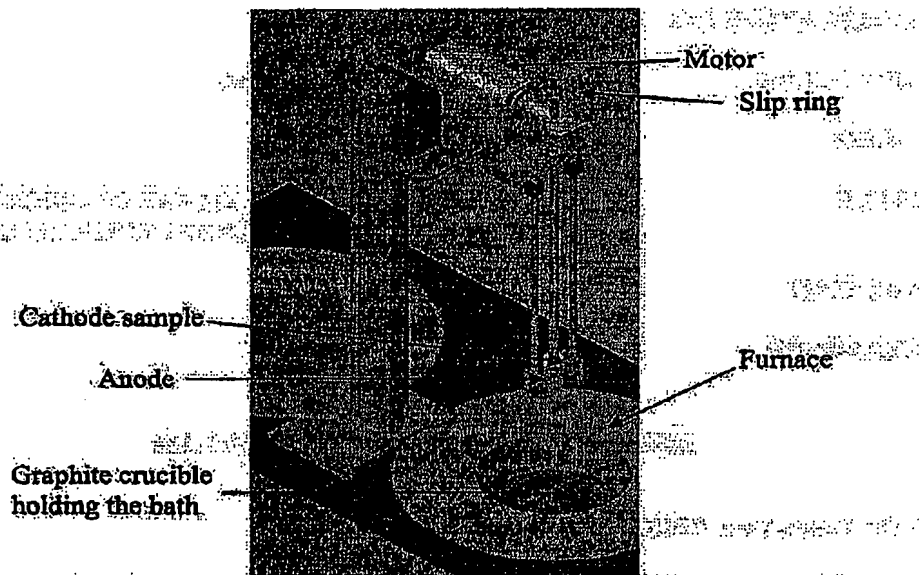


Fig. 1. Test apparatus

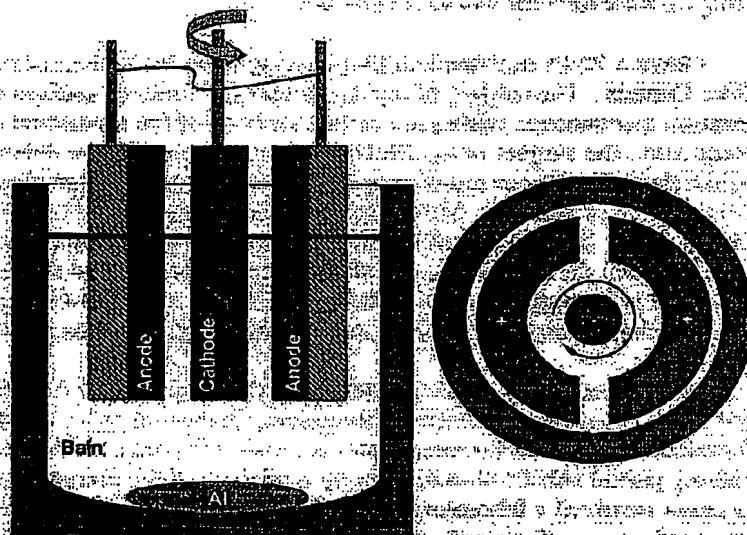


Fig. 2

The test apparatus could be rotated at speeds between 30 and 150 rpm but, in this experiment, the samples were rotated at 115 rpm and electrolysis was carried out for 100 hours in an electrolyte having the following composition:

Cryolite (Na_3AlF_6) : 76.8%wt
 AlF_3 : 11.2 %wt
 CaF_2 : 6%wt
 Al_2O_3 : 6%wt

The bath ratio (mass NaF / mass AlF_3) was therefore 1.1 (it is 1.5 for pure cryolite).

The bath temperature was 965°C and the cathode current density was approximately 0.8, which is close to what is used in industrial cells.

The rotation of the sample simulates the movements of the fluids relative to the cathode surface in an electrolysis cell, where magneto-hydrodynamic forces drive the metal and the bath. The moving fluids accelerate mass transport from the cathode to the bath and thus it accelerates the wear of the cathode. In order to simulate the real phenomena occurring in a cell, one thus needs to generate this relative movement of the fluids by artificial means (here, rotating the cathode part).

4. Two cathode materials were tested in this way, namely:

TBA: A carbon-based composite (anthracite with a pitch binder) containing 50% TiB_2 by weight.

TBAa: The same carbon-based material containing 35% TiB_2 and a stoichiometrical mixture of $\text{TiO}_2/\text{B}_2\text{O}_3$ equivalent to 1.5% by weight.

5. The results are shown in Exhibit A attached hereto, which shows photographs of the TBA and TBAa materials (as so indicated) after the tests. The mixture containing partial substitution with the oxide mixture (TBAa) shows superior erosion resistance (i.e. 1 mm of erosion compared to greater than 5 mm of erosion for the TBA material).

6. I conclude from this that oxide additives, when substituted in part for TiB_2 in the cathode material, provide a better wettability of the materials by aluminum in these conditions. This leads to a better protection of the materials against bath corrosion and thus to a lower wear.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

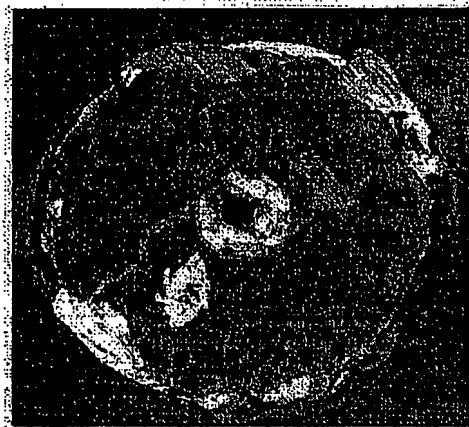

Dr. Pierre-Yves BRISSON

Date:

2007-09-20

EXHIBIT A

TBA



Measured Erosion > 5 mm

TBA



Measured Erosion = 1 mm